Development of a New Environmental Scanning Probe Microscope: JSPM-5400

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Introduction

The Scanning Probe Microscope (SPM), including the Atomic Force Microscope (AFM) and the Scanning Tunneling Microscope (STM), is widely used as a powerful tool to measure a topographic shape of a sample with nanometer-scale resolution. Recently, applications of the SPM to study of material properties have been increasing. The reason for this is that in addition to the original topographic-measurement capability, the SPM has provided increasingly versatile capabilities, such as visco-elasticity atomic-force microscopy (VE-AFM), lateral-modulation friction force microscopy (LM-FFM) and scanning Kelvin probe microscopy (SKPM), which enable one to obtain mechanical or electrical properties of the sample with its topography.

Most of the modern SPM instruments are used at ordinary temperatures and pressure. There are, however, strong demands to use SPM in actual environments, such as at low or high temperatures, in controlled atmospheres and in vacuum, in which observed samples are practically used as industrial products. This situation has necessitated the development of a SPM in which sample environments can flexibly be controlled.

To satisfy the demand, JEOL has developed a new environmental SPM: the JSPM-5400. This innovative SPM is aimed at achieving multi-functional measurements and flexible sample environment control as well as providing high-resolution imaging.

Features of JSPM-5400

Figure 1 shows the external appearance of the JSPM-5400. The JSPM-5400 has been developed on the basis of the following three main concepts.

High-resolution imaging without damage using Non-contact AFM (NC-AFM)

Usually, the AFM has been used with amplitude detection. As for this detection method, the cantilever is scanned with intermittent contact on the sample surface. Therefore, there is always a possibility for the cantilever to damage it, especially when the soft sample is measured.

JEOL SPMs are equipped with a Non-contact AFM (NC-AFM) that employs a constant excitation amplitude FM (frequency modulation) detection method (JEOL patent). The main feature of the NC-AFM is to provide a high-resolution topographic image without contact between the apex of the cantilever and the sample surface. The sensitivity of this FM detection has further been improved due to the employment of a digital PLL (Phase-Locked Loop) for a new amplifier, which enables high-stability imaging with atomic-level resolution. This robust capability is demonstrated in a topographic image of mica obtained with the NC-AFM (Fig.2). Because an auto gain control (AGC) circuit is incorporated in the new amplifier with a constant amplitude mode, the JSPM-5400 enables us to observe a dissipation image of a sample, which displays energy dissipation in the sample.

Environmental control for versatile imaging

To achieve versatile imaging under diverse sample environments controlled by the JSPM-5400, drastic improvements of capabilities and extension of optional accessories have been made.

Flexible environmental control

The vacuum quality has increased due to the...
improvement of the structure of the SPM head and the employment of an optional cold trap system. By the use of the cold trap system, the vacuum pressure around the sample reaches down to $8.5 \times 10^{-6}$ Pa, when the pressure is approximately $2.0 \times 10^{-5}$ Pa with ordinary pumping. Thus, gas adsorption on the sample can be reduced to a minimum, maintaining a clean sample surface. Because optical-axis alignments of the laser and photo detector are designed to be performed from the outside of the vacuum, the JSPM-5400 provides optimal imaging for any cantilever.

**Extension of optional accessories**

The JSPM-5400 can be equipped with extended optional accessories for multi-purpose, multi-functional measurements, including heating/cooling holders, a temperature controller for stable observation of heated/cooled samples and an airlock sample exchanger that enables a sample to be changed while maintaining the vacuum.

**New technologies for safety and easy operation**

The JSPM-5400 has served extended new functions, such as shortening measurement and approach time, and preventing collision between the sample and the cantilever.

**Safety high-speed approach**

In a conventional SPM, the cantilever approaches the sample at a constant speed and stops at a specified position (Fig. 4(b)). Thus, when the distance between the cantilever and the sample is far, approaching requires a long time.

To shorten the approach time, the JSPM-5400 uses a two-step, safety high-speed approach: high-speed approach and fine approach (Fig. 4(a)). The former detects weak attractive force from a far field, whereas the latter detects the distance between the cantilever and the sample while finely controlling the sample position; therefore, the cantilever can approach the sample in a shorter time. Figure 5 shows a comparison of the approach time with the conventional constant-speed approach and with that with the safety high-speed approach, indicating that the safety high-speed approach is much faster, reducing the approach time significantly.
speed approach shortens the approach time by about one sixth compared to the conventional constant-speed approach.

The safety high-speed approach is effective for highly light scattering samples and transparent samples, which are difficult to know the actual distance between their surfaces and the cantilever.

**Damage-less scan control**

Damage-less scan control is used to operate the probe within a limited area (distance L in Fig. 6) with respect to a reference height (point A in Fig. 6). In the observation of the edge of cross sections, which is difficult for the conventional SPM, the probe can be safely scanned without collision of the apex of the cantilever with the edge. In addition, the damage-less scan control incorporates a reference-height automatic renewing function. This function automatically renews the reference height in the upper direction of a concave portion even for a sample with steep concavity (patent applied for). Thus, the JSPM-5400 can measure samples with unknown topographic shapes, without damage.

Figure 7 shows a topographic image of a multi-layer optical film using the damage-less scan control. The layer structures of the multi-layer optical film can be observed successfully even around the edge.

**Fast and simple positioning**

A sample of a polymer mixture tends to have components with different diameters, causing steep unevenness of the surface of the sample. This may lead to the situation that the cantilever collides with the sample surface and fast observation to obtain its entire image is difficult.

Combined use of the skip-scan function and damage-less scan control incorporated in the JSPM-5400 enables us to rapidly observe the entire image of the sample having an unknown topographic shape, without the collision between the cantilever and the sample. Figure 8 shows a topographic images of glue made of a polymer mixture, obtained by a combination of the skip-scan and the damage-less scan control. In addition, use of a zoom function for the field of interest, which allows us to arbitrarily specify the ratio of horizontal and vertical sizes and rotations, can dramatically shorten useless scan time.

**Probe auto tracking**

This function automatically corrects the vertical drift caused by thermal expansion or contraction of the sample during observation with heating or cooling, preventing the apex of the probe from crushing or getting away from the sample surface over a long period of time. Figure 9 shows images of a polyester-based compound resinous film, sequentially obtained using probe auto tracking. Topographic and phase images were acquired through temperature variations where the sample was heated in steps of 1°C per minute.

No change is seen in the topographic image, but significant changes are detected in the phase images of the surface. The phase images reveal the change of the sample state, which becomes uniform by heating.

**Digital zoom**

Up to 2048 × 2048 pixels are provided for images, allowing images to be simultaneously displayed for up to 5 channels. Figure 10 shows phase images of a two-component wax mixture, which are displayed with maximum pixels. Since the phase image contains information on physical properties of materials, it is frequently utilized simultaneously with the topographic image. From images acquired with maximum pixels, we can easily pick up enlarged images on areas of interest in digital zoom.

**Summary**

This paper has introduced the JSPM-5400, which achieves flexible environmental control, high-resolution imaging and ease of use. This versatile environmental SPM is especially suited for high-resolution observations of polymer samples, which will give deeper insights for various fields including the development of new products.
Fig. 9 Topographic images (upper) and phase images (lower) of a polyester-based compound resinous film through temperature variations (scan size: $2 \mu m \times 2 \mu m$).

Fig. 10 Phase images of a two-component wax mixture.